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# **Final Report**

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### Fiber Pulling Apparatus

by

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#### 1.0 INTRODUCTION

The fiber optics industry has grown into a multi-billion marketplace that will continue to grow into the 21st century.[1-5] Optical fiber communications is currently dominated by silica glass technology. Successful efforts to improve upon the low loss transmission characteristics of silica fibers have propelled the technology into the forefront of the communications industry. However, reaching the theoretical transmission capability of silica fiber through improved processing has still left a few application areas in which other fiber systems can provide an influential role due to specific characteristics of high theoretical transmission in the 2 - 3 micron wavelength region.

One of the other major materials used for optical fibers is the systems based upon heavy metal fluoride glass (HMFG). Commercial interest is driven primarily by the potential for low loss repeaterless infrared fibers. An example of the major communications marketplace which would benefit from the long distance repeaterless capability of infrared fibers is the submarine cables which link the continents.[5]

When considering commercial interests, optical fiber systems provide a healthy industrial position which continues to expand.[1-6] Major investments in the systems used for optical fiber communications have continued to increase each year and are predicted to continue well into the next century. Estimates of 8.5% compounded annually are predicted through 1999 for the North American market and 11% worldwide. The growth for the optical fiber cable itself is expected to continue between 44 and 50 per cent of the optical fiber communications budget through 1999. The total budget in 1999 world-wide is expected to be in the neighborhood of \$9 billion.[3] Another survey predicts that long haul telecommunications represents 15% of a world-wide fiber optics market in 1998.[4] The actual amount allotted to cable was not specified. However, another market research had predicted that the cable costs alone represents more than 50% of the total budget each year through 1998.[2]

A newly emerging activity is the commercial development of doped optical fibers which can be pumped by laser diodes to provide amplification of the communication signals.[6] This technology is newly emerging and will be developed for commercial interests in the United States by Galileo Electro-optical Incorporated in Sturbridge, MA on a license from British Telecom. Long repeaterless communication links provide the biggest stimulus for this technology. As an example of the of the revenues involved in the optical fiber communications

industry, the current trade journal lists that for the fiscal years, 1991 - 1994, 185 separate undersea links were established. In addition, another 105 links are planned through 1998. The distribution of revenues involved in the undersea installations is roughly \$8.5 billion through 1993 and another \$13 billion planned through 1998. A large portion of the future activity (34%) is planned for Southeast Asia and the Pacific Region. Other examples of the commercial utility of optical fiber networks is given in a recent scientific symposium in which the outlook for HMFG infrared fibers was determined to be very bright.[7]

Another area of interest lies in the use of fiber optics for laser surgery delivery systems, in which an optimal match between laser wavelength and fiber transmission characteristics occurs. For precise removal of tissue during surgery, research has shown that a wavelength in the 2.5 - 3.0 microns performs best. Experience with the combination of a pulsed Er:Yag laser (2.9 microns) delivered through a ZBLAN fiber has shown that this combination allows the removal of both fibrous and heavy calcified arterial plaque with little or no signs of thermal damage.[8] The 2.9 micron radiation corresponds quite closely with maximum tissue absorption (about 20 times greater than the 10.6 radiation from carbon dioxide lasers) and consequently allows very small penetration depth and precise tissue removal with no charring. This activity has proven to be of commercial value to small entrepreneurial companies such as Infrared Fiber Systems Inc. in Silver Spring, MD.

Process improvements which can enable heavy metal fluoride fibers to meet the their theoretical capabilities will provide the communication and medical industries with very desirable technology and products. Current manufacturers are very small and growth would be expected, as well as technology spin-offs to other manufacturers. It is the goal of space based experiments to provide much higher quality fiber with near theoretical transmission capability for development of commercial markets in the United States.

#### 2.0 BACKGROUND

The initial research into the use of multicomponent fluorides based on ZrF<sub>4</sub> chemistry led to the 1974 discovery of an amorphous product by Professor M. Poulain and his co-workers a the University of Rennes in France.[9] The remarkable feature of this work was that all the starting materials were crystalline and with the proper stoichiometry, amorphous glasses could be

produced. From this work, and others beginning to take an interest in the United States, the chemistry of the heavy metal fluoride glasses began to take shape.

An example of the HMFG chemistry is shown in Figure 1 below. The phase diagram for the ZrF4<sub>4</sub>-BaF<sub>2</sub>-LaF<sub>3</sub> (or ZBL glass) shows a region in which stable glass and a region of unstable glass devitrifies into crystalline material. An understanding of this chemistry led to the conclusion that ZF<sub>4</sub> was the glass former, BaF<sub>2</sub> was the modifier and a small amount of LaF<sub>3</sub> helped to decrease the devitrification rate was called a stabilizer. Other HMFG compositions were tried in the 1980's but none were as prolific in the research laboratories in both Europe and the United States as the ZBL systems. Further research evolved into the material of interest today, ZBLAN, in which AlF<sub>3</sub> and NaF are added to improve the glass forming capabilities of the final product. Current industrial practices uses hafnium for the cladding and consequently HfF<sub>4</sub> has also become an important ingredient in the overall chemistry.

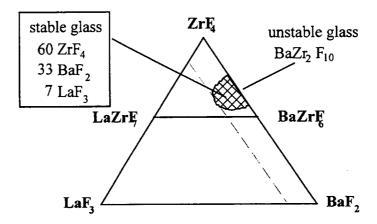


Figure 1. Phase diagram of typical HMFG system.

The primary focus on HMFG as an optical fiber material, in spite of its early shortcomings in reliable glass forming due to devitrification, was the theoretical prediction of low-loss of 0.001 dB/km at 3 microns as compared to silica (0.2 km/dB at 1.55 microns). The original theoretical work which indicated that the HMFG materials would have very good transmission characteristics was published by S. Shibata, et. al. and extended in more depth by the work of Lines, however equations defined in his work show the following result in Figure 2.[11]

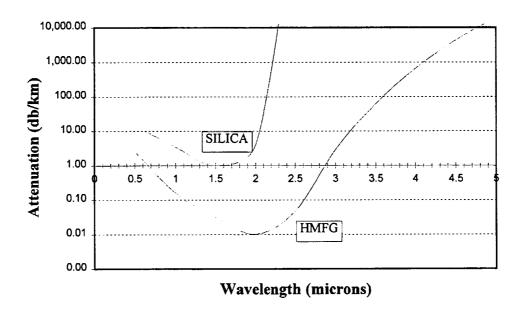


Figure 2. Comparison of theoretical attenuation for HMFG with average grade silica fibers.

The lower loss potential for long distance repeaterless fiber optic cable systems (undersea) and special purposes (medical laser surgery) promised a potential market of a high magnitude. For this reason many industrial and government laboratories world-wide began research in the HMFG including, Nippon Telegraph Telephone, British Telecom Labs, Naval Research Labs, Rome Air Development Center, ATT Bell Labs, Furukawa Industries, Corning Glass, Centre National d'Etudes des Telecommunications, and many more.

A number of formulations have been used to fabricate optical fiber devices in the 1980's and 90's. The use of zirconium fluoride as the primary constituent with fluorides of other metals such as barium, aluminum, sodium, lanthanum, etc. has led to general fiber compositions such as illustrated in Figure 3 below. The acronym used to describe these materials is based upon the metallic elements used in the fiber.

Acronym	ZrF <sub>4</sub>	$BaF_2$	LaF <sub>3</sub>	$AlF_3$	NaF	$T_{g}$	$T_{x}$	$T_c$
ZBLAN20	53	19	5	3	20	263	384	405
ZBLAN8	55	30	3	4	8	295	381	401
ZBLA	60	30	3	5		312		384
ZB	65	35				295	352	365

Figure 3. Typical formulations used for HMFG optical fibers.[12]

The major difficulties in ZBLAN processing have been determined to be due to the development of microcrystallites during both preform casting and during draw.[13-16] A major improvement in both the transmission and strength of the fiber can be obtained through a fiber fabrication process in which the microcrystallite population is substantially reduced.

Several research activities in microgravity science have observed the decreasing tendency of crystallization while processing in the microgravity environment.[18,19,21] The rationale for this behavior has not been determined at this time and continues to be a concept requiring further research. In the meantime, the industrial uses for an improved ZBLAN fiber provides a need to obtain a microgravity processed material in order to provide a better product of both industry and medicine.

#### 3.0 CONTINUING WORK WITH THE GLASS ANNEALING FURNACE

In February of 1997 efforts turned to reflying the KC-135 Glass Annealing Furnace (GAF) as part of the continuing research efforts into the crystallization of ZBLAN fibers. During the previous flights of the GAF the hardware package was mounted in a manner in which the long axis of the fiber sample was perpendicular to the gravity vector. In many cases (during previous flights) this seemed to cause the fiber to "stick" to the inside wall of the sample ampoule and thus interfere with the results. Figure 4 clearly shows the effect on the ZBLAN

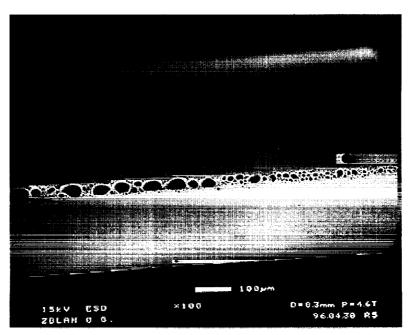


Figure 4: Surface reaction effects of ZBLAN with silica.

fiber reacting with the silica in the ampoule wall. This reaction causes the pitting as seen in the SEM photograph. To help alleviate this problem for the upcoming flight the package was remounted 90 degrees from its original position. This would allow the fiber a greater chance of avoiding full contact with the ampoule wall during the high-g period of the maneuver. Because this was considered a major change in the hardware the necessary safety documentation had to be prepared and submitted to JSC. No problems were encountered during this paperwork phase.

### 3.1 GAF Flight and Ground Results

During the week of April 21, 1997, the GAF was flown on NASA's KC-135 to process samples provided by Dr. Rifik Kortan of Lucent Technologies. Dr. Kortan was responsible for all sample preparations, which included drawing the ZBLAN fiber and loading the samples into the quartz ampoules.

During the first flight on April 22 no samples were processed. Only calibration tests were performed to verify the thermal characteristics and stability of the furnace in the various gravity levels. The results of these tests in the following table provided the verification that the furnace thermal characteristics were stable. Test Series 1 indicates the thermal stability of the preheat zone during the change from high-g too low-g.

TEST SERIES 1: PREHEAT ZONE STABILITY				
DURING CHANGING G-LEVELS				
PARABOLA	TEMPERATURE IN	TEMPERATURE AT		
NUMBER	HIGH-G	END OF LOW-G		
1	255.1 C	254.3 C		
2	254.2 C	254.5 C		
3	254.2 C	253.9 C		
6	254.7 C	253.8 C		
7	254.3 C	254.2 C		
8	253.6 C	254.7 C		

Test Series 2 indicates the change in temperature the sample experienced during the transition from a stable temperature in high-g to the temperature reached at the end of low-g.

TEST SERIES 2: TRANSITION FROM PREHEAT  ZONE TO ANNEALING ZONE						
DADADOLA						
PARABOLA	TEMPERATURE IN	TEMPERATURE AT				
NUMBER	HIGH-G	END OF LOW-G				
9	254.3 C	356.6 C				
11	252.4 C	355.3 C				
14	252.8 C	362.4 C				
16	251.3 C	358.3 C				
24	253.4 C	361.6 C				

Test Series 3 indicates the thermal stability of the annealing zone furnace during the change from high-g to the end of low-g. As indicated by the table the thermal stability is remarkably constant.

TEST SERIES 3: ANNEALING ZONE STABILITY DURING CHANGING G-LEVELS					
PARABOLA	TEMPERATURE IN	TEMPERATURE AT			
NUMBER	HIGH-G	END OF LOW-G			
17	363.3 C	363.4 C			
18	363.5 C	363.4 C			
25	362.1 C	362.3 C			

During all the thermal tests the preheat zone setpoint was at 240°C and the annealing zone setpoint was at 350°C. A fine gauge type K thermocouple placed inside an empty quartz ampoule was used to collect the thermal data and therefore simulated the thermal mass of a loaded quartz ampoule.

On the following day, during the second KC-135 flight, a total of nine ZBLAN loaded ampoules were processed in the GAF. Prior to processing the first ampoule the first five parabolas were used to calculate an average low-g time period. From the start of low-g to the end was found to be 24.3 seconds. This number was used to determine the maximum soak time of 21 seconds for the sample while in the annealing zone furnace. The initial preheat and annealing zone temperature setpoints were set to 240°C and 350°C, however the annealing zone temperature was reduced to 345°C after processing the first sample. It was determined that the sample melted too much at 350°C. The 345°C setpoint was used for all of the subsequent processing runs. The following table provided a summary of the flight days events.

PARABOLA NUMBER	SAMPLE I.D.	NOTES
8	ZBLAN-302-1, #1	Sample melted too much, setpoint to 345, approx. 3 min. preheat
12	ZBLAN-302-1, #2	Sample looked good, approx. 9 min. preheat period
16	ZBLAN-302-2, #1	Sample looked good, fiber stuck to ID wall, approx. 2.5 min. preheat
20	ZBLAN-302-2, #2	Sample looked good, approx. 2.7 min. preheat period
22	ZBLAN-302-3, #1	Sample looked good, approx. 3.7 min. preheat period
26	ZBLAN-302-3, #2	Large melt droplet on end of fiber, approx. 2.5 min. preheat period
30	ZBLAN-302-3, #3	Sample looked good, approx. 2.6 min. preheat period
32	ZBLAN-302-4, #1	Sample looked good, fiber stuck to ID wall, approx. 2.1 min preheat
36	ZBLAN-302-4, #2	Small fiber diameter, approx. 3.0 min. preheat period

On the following day, April 24, another seven samples were processed in the GAF system. Again the first five parabolas were used to calculate the average length of low-g which was determined to be 24.3 seconds. The annealing zone soak times were therefore limited to 21 seconds as before. Sample ID ZBLAN-302-4, #2 was processed twice, once on April 23 and again on the 24<sup>th</sup> by accident. The last sample, ZBLAN-302-6, #3 was processed three times during three successive low-g periods. The sample was retracted back into the preheat zone just prior to entering high-g.

PARABOLA NUMBER	SAMPLE I.D.	NOTES
9	ZBLAN-302-4, #2	Processed twice (once yesterday), approx. 2.4 min preheat period
13	ZBLAN-302-5, #1	Sample looked good, approx. 2.6 min. preheat period
18	ZBLAN-302-5, #2	Sample looked good however fiber stuck to ID wall, approx. 4.5 min PH
21	ZBLAN-302-5, #3	Sample looked good, approx. 4.0 min. preheat period
25	ZBLAN-302-6, #1	Sample looked good, approx. 3.0 min. preheat period
29	ZBLAN-302-6, #2	Sample looked good, approx. 3.0 min. preheat period
35, 36, 37	ZBLAN-302-6, #3	Processed three times, fiber reformed into two small BB's, approx. 10 mi preheat total

Ground runs were preformed in July 1997, with a total of six samples processed. All conditions (with the exception of gravity) were the same as those used during the flight runs. The preheat zone soak times were held constant to 3.0 minutes for each sample and the soak times in the annealing zone were held to 21 seconds. Only one sample was not processed either as a flight or ground sample and was used as a control or "as received" sample.

The following microphotographs provide a representative comparison between the flight and ground processed samples. The ampoules were broken open to remove the ZBLAN fibers for better photography. After photographing they were then replaced into the ampoules and taped closed. The fibers were photographed with polarized light to highlight surface crystals at a magnification of 56X.

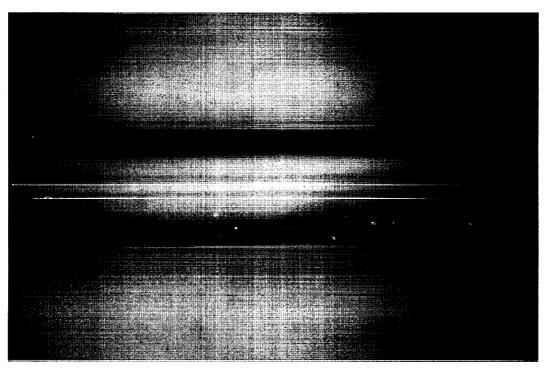


Figure 5: "As-received" control ZBLAN sample – not processed. The small white particles on the surface of the fiber are ambient dust and ZBLAN particles created when breaking the fiber to the proper length.

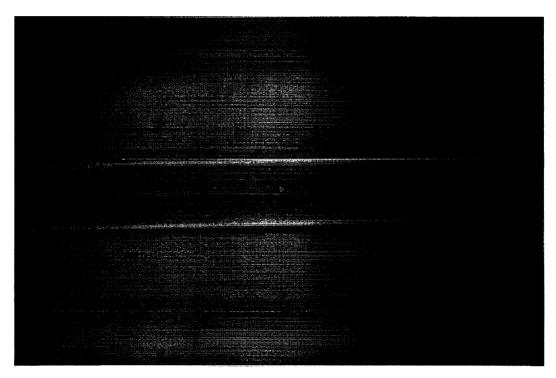


Figure 6. Ground processed sample ZBLAN-302-1, #3. Notice the "frosted" appearance of the fiber surface.

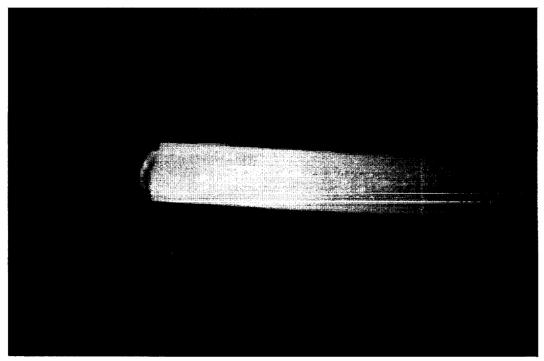


Figure 7. Another example of a ground processed sample ZBLAN-302-6, #5. In this case the surface crystallization is even more prevalent

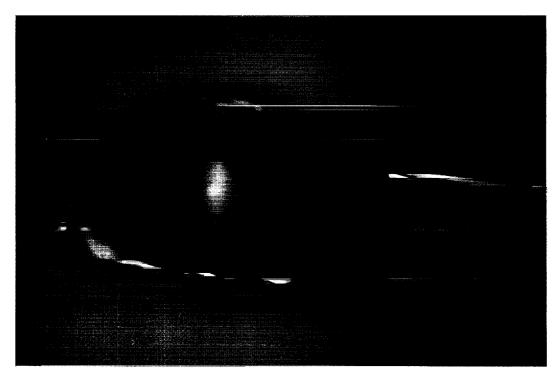


Figure 8. Flight processed sample ZBLAN-302-1, #1. Notice the lack of any evident surface crystallization. This sample is representative of all the flight processed samples.



Figure 9. Another view of flight processed sample ZBLAN-302-1, #1

#### 3.2 Conclusion of GAF Results

During the April 1997 KC-135 flight of the GAF system some important information was obtained in regards to the thermal consistency of the two furnaces in low-g vs. high-g. In reviewing the thermal data it appears that there is little if any change in the thermal profile of either the preheat or annealing zone furnace when comparing the high-g data to the low-g data. This is important in that it indicates the ZBLAN fiber sample experiences the same thermal conditions whether it is processed in low-g or high-g. Since the thermal measurements took place over the gravity range of high and low is can be assumed that the thermal consistency is the same between low-g and normal-g. It can therefore be concluded that the crystallization phenomenon, or lack thereof, is not due to differences in the thermal processing parameters (i.e. convection) in the GAF but due to the differences in gravity.

The presented microphotographs provide good correlation to previous GAF experiments results [22, 23]. It can be clearly seen that ZBLAN samples processed in a normal 1-g environment develop microcrystallites on the surface of the fiber where as flight processed samples in low-g did not. In the ground processed samples (six in all) two of the samples (ZBLAN-302-1, #3 and ZBLAN-302-6, #5) showed almost complete surface crystallization. The remaining four ground samples processed had only about 5 to 10 percent crystallization. The reason why is as yet undetermined at the time of this printing. It is suspected that differences in how the samples were prepared and sealed inside the ampoules may be the reason. Work is currently underway at UAB to provide SEM and TEM microphotographs of the samples presented in this report.

### 4.0 SUPPORT EFFORTS ON THE PPF PROTOTYPE

In the spring of 1997 the MSFC Space Sciences Labs began development of a Preform Processor Furnace prototype. This furnace is intended as an engineering test bed for developing the Shuttle MidDeck version. The purpose of the PPF is the processing ZBLAN preforms in the microgravity environment of earth orbit. In view of the results obtained from the GAF experiments it is a strong possibility that earth based manufactured ZBLAN preforms which contain microcrystallites can be reheated to 800°C in microgravity, dissolve out the defects, and then quench the preform back into a solid rod. The preforms would then be returned from space and distributed to the industrial partners who would then draw the ZBLAN fiber. It is expected that by starting out with a preform free of microcrystallites a nearly perfect optical fiber can be produced.

General design support was provided on a weekly basis in the development of PPF prototype furnace. This support included identification of temperature and power control subsystems and parts which could be readily obtained from commercial sources and subsequently used (with minor modifications) in the actual flight hardware system. Electrical schematics were developed and provided to the SSL team in charge of assembling the prototype system. Copies of these drawings are included in Attachment 2 at the end of this document. In addition, other areas of general support were also provided including thermal design assessments, Shuttle and astronaut interface requirements, safety, sample cartridge designs, and data acquisition/control issues.

#### 5.0 EARLY DEVELOPMENT OF MAGE

In addition to the GAF activities an additional task was taken on to develop a Space Shuttle experiment which could prove that ZBLAN could be held in a magnetic field and then processed in the same manner as GAF samples. In this way ZBLAN could be processed in a containerless environment and over a longer period of time. This would provide additional data points into the growth kinetics of the microcrystallites. Early in October of 1997 work began on developing such an experiment for flight on STS-95 in October 1998. Prior to that, a simpler demonstration package was developed for testing out the theory on the KC-135. This was a collaborative effort between UAH and Boeing. The concept for the levitating the ZBLAN (or any other diamagnetic material) was developed by Boeing [24].

The purpose of MAGE (Magnetic Glass Experiment) was to demonstrate the levitation of ZBLAN glass in a magnetic field. ZBLAN glass, like many other materials, is diamagnetic. In the absence of gravity this property allows materials to be suspended in a magnetic "bottle" between two opposing permanent magnets.

#### 5.1 MAGE Experiments on the KC-135

Experiment hardware was designed to demonstrate magnetic levitation on board the NASA KC-135 reduced gravity aircraft. Two separate weeks of flights were made. Each flight week was four days, with about 40 parabolas each day. Each parabola provided about 25 seconds of reduced gravity. To further reduce the gravity level, the experiment package was allowed to free float at the end of an umbilical consisting of power and data cables. The free float resulted in brief periods of low gravity on the order of 1 to 2 milli-g. These lasted from 2 to 5 seconds. A camera on the free float package allowed a video tape record of each flight.

The first flight week was July 12 to 17, 1997. The magnet/sample holder assembly that was flown is shown in figure 10.

The sample containers for this flight were glass cylinders attached to the end of the sample holders. Two sample materials were flown: ZBLAN glass and Bismuth. Bismuth was chosen because it is the most diamagnetic material and therefore is most affected by the magnetic field. The samples were in an air environment within the sample container. During the first flight day it was observed that if the samples were subjected to an initial disturbance they never became stable within the container. Any small motion resulted in the sample bouncing off

the walls of the container similar to a "ping pong" effect. Another problem was the adhesive used to seal the containers did not fully cured and as result remained slightly tacky. This allowed the samples to stick in the corners. These problems resulted in the early withdrawal of the experiment from the aircraft after two days.

The second KC-135 flight took place the week of December 8, 1997. The hardware was redesigned to accommodate larger, more powerful magnets. The magnet holder was made from acrylic blocks. Two one inch diameter by ½ inch thick magnets and two-½ inch diameter by ¼ inch thick magnets were mounted on either side of a gap that formed the magnetic "bottle". A drawing of the magnet holding assembly is shown in figure 11. Clear plastic sample containers were used for the samples in air. These had a foam pad at one end for damping. Plastic vials were used for containers in which a liquid solution was used to provide viscous damping. The best results were obtained with this approach. The ZBLAN was suspended between the magnets on several occasions for short durations. The undisturbed levitation time was on the order of two to three seconds.

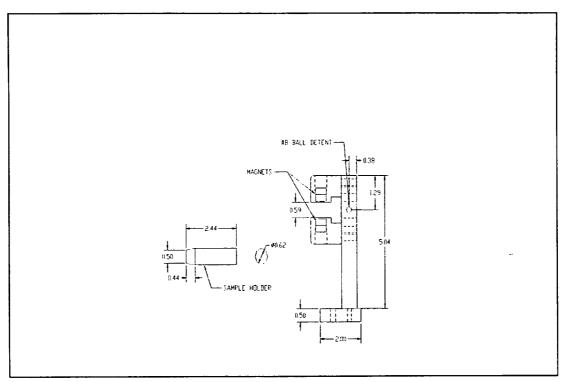


Figure 10: Side view of first prototype magnetic levitator (mirror not shown).

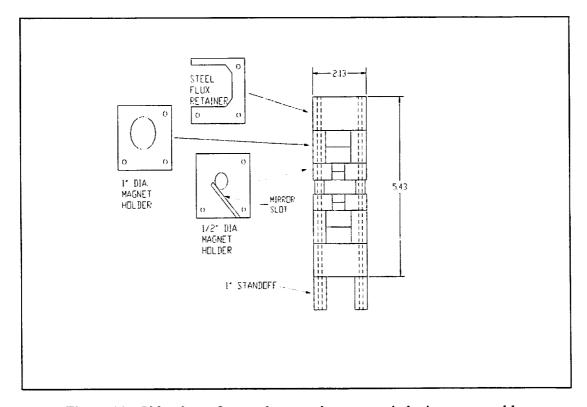


Figure 11: Side view of second generation magnetic levitator assembly.

### 5.1.1 Summary of Accelerometer Data from MAGE

The following accelerometer data is a representative sample of typical free float periods for the MAGE hardware during KC-135 low-g maneuvers. Each data point is an average of

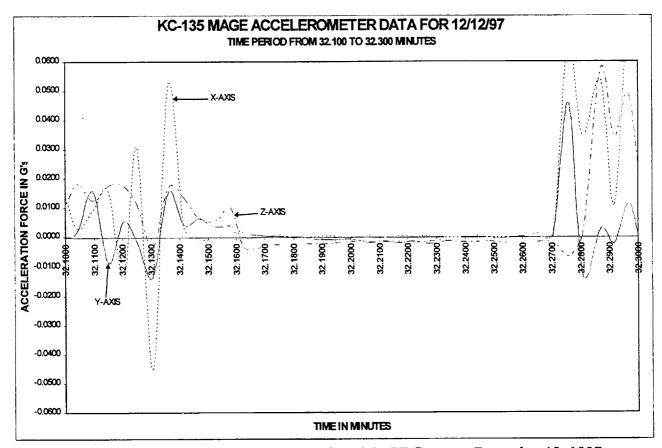


Figure 12. Three axis accelerometer plot from MAGE flown on December 12, 1997

1000 readings which filters out almost any EMI noise present on the accelerometer signals. This data was collected during the December 12, 1997 KC-135 flight day. The following captured video images (Figures 13 and 14) are of a ZBLAN sample being held within the magnetic bottle during the low-g period shown in the above graph. The ZBLAN sample was rectangular shaped 1.5x1.5x3 mm in size. The sample cell was filled with an ethanol/MgCl mixture that had the same magnetic properties as air.

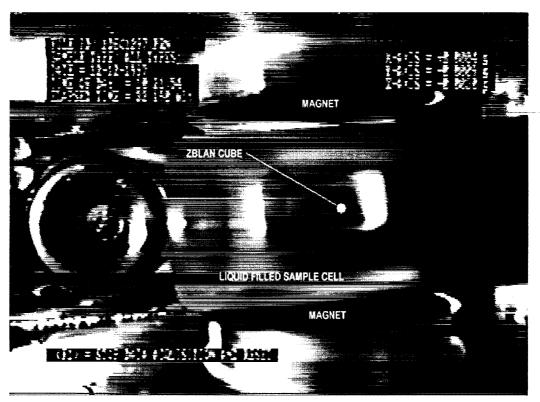


Figure 13. Video image of a ZBLAN cube suspended in the magnetic bottle region of MAGE.

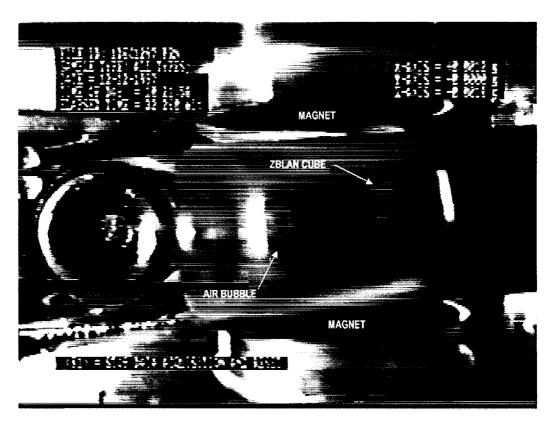


Figure 14. Image of the same cube floating out of the magnetic bottle region due to the negative 0.002 milli-g acceleration in the Z-axis.

#### 5.2 Shuttle/SpaceHab Glovebox Experiment

The previous experiments were precursors to designing an experiment to fly on the Shuttle on STS 95 scheduled for October 1998. The experiment is being designed to be carried out in the Microgravity Glovebox (MGBX) to be mounted in the SpaceHab module. A visit was made to the MGBX facility at Marshall Space Flight Center (MSFC) to become familiar with the features and accommodations offered. There are two items supplied that can be utilized: a video camera and a light source. These items and others were measured to allow inclusion in the design. The measuring was necessary since there are no dimensioned drawings available for the Glovebox or its accessories. An Interface Control Document (ICD) has not been developed, so users must make their own measurements and obtain other information on connectors, power sources, etc.

A conceptual design of the hardware has been completed. A set of the drawings is attached. A request has been made be Boeing to include additional equipment and temperature sensors to perform measurements of magnetically driven convection of heated air. These requirements have not yet been incorporated into the design.

#### 5.2.1 Materials List

The following are the materials that have been identified for use in the Shuttle experiment hardware. The list should not be considered to be complete or final.

- 1. Aluminum
- 2. Flight approved polycarbonate
- 3. Stainless steel
- 4. Sheet steel (magnetic shielding)
- 5. Silica glass (sample container)

#### 5.3 Attachment 1

This is the Sample Material Submittal that was sent to Amy Haas, the experiment Payload Coordinator at SpaceHab (STS-95) on 11/17/97. This information was provided as part of the initial safety documentation.

**Experiment:** Magnetic Levitation of ZBLAN Glass (MAGE)

PI: Dr. Dennis Tucker, NASA, 205-544-2685

Co-PI: Dr. Brian Tillotson, Boeing, 253-773-4547

Hardware development: Leonard Adcock, UAH, 205-890-6086 x 225

**Experiment Summary:** The experiment will be carried out in the Glovebox. ZBLAN glass is diamagnetic. In the absence of gravity it can be levitated magnetically using two sets of permanent magnets. The magnets are mounted opposed with a gap of approximately one half inch between them. The magnets are held in a holder designed for the purpose. The samples, in their containers, are inserted into the gap and observed. The observation will be made using a video camera, recorded, and downlinked if possible.

#### **Test Materials:**

Material	Quantity	Weight	Volume
ZBLAN* glass	6	<10g ea.	~25 mm³ ea.
Silica glass	1	<10g ea.	~25 mm³ ea.
Lysozyme protein crystal in aqueous solution of NaCl	1	<10g ea.	~25 mm³ +2cc.
Bismuth	1	<10g ea.	~25 mm³ ea.
Water	1		~2cc
Wood	1	<10g ea.	~25 mm³ ea.
Plastic	1	<10g ea.	~25 mm³ ea.

<sup>\*</sup> ZBLAN is a heavy metal fluoride glass containing Zirconium fluoride, Barium fluoride, Lanthanum fluoride, Aluminum fluoride, and Sodium fluoride.

Weight of Materials: see table above.

Test Conditions: Five to ten minutes of video recording of each sample.

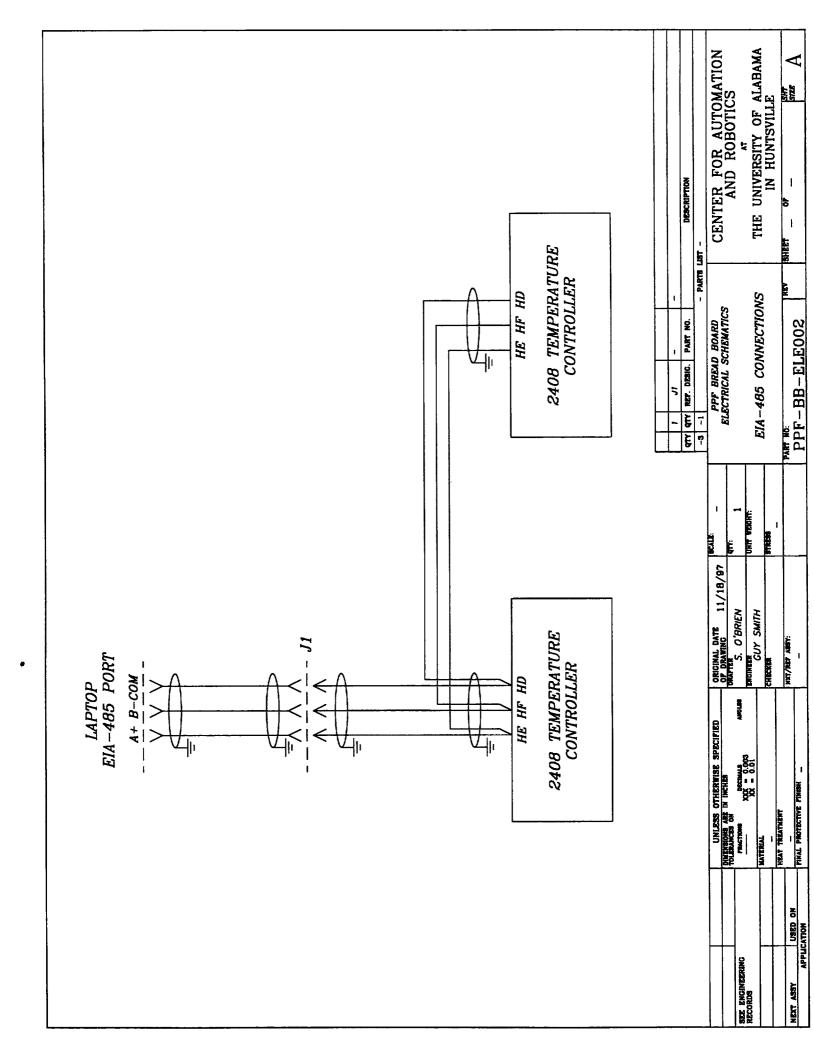
Number of Samples: 11

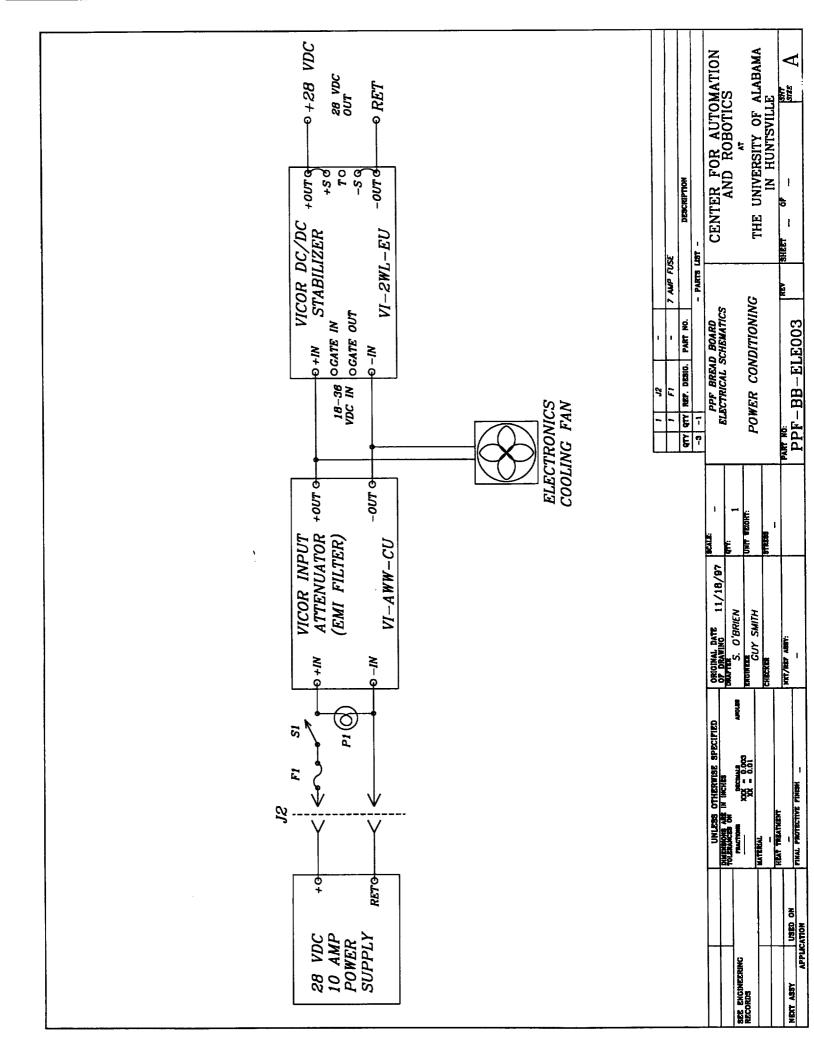
#### Types and Estimated Level of Toxic Hazard:

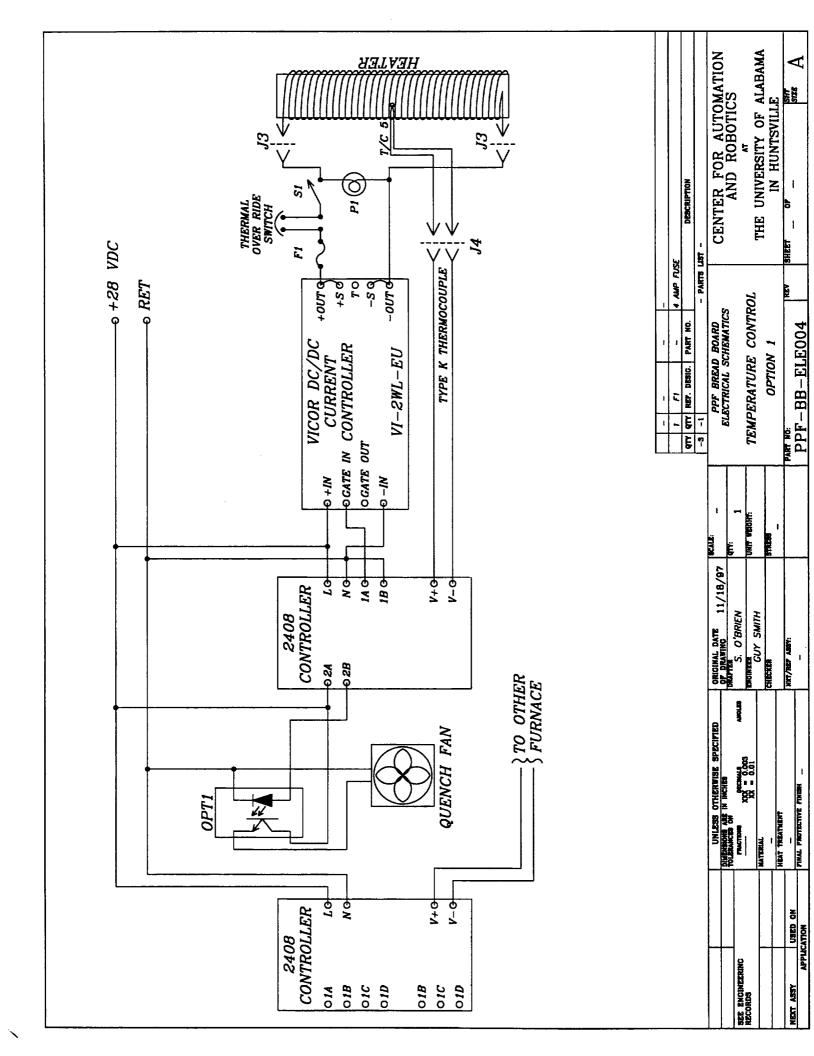
**Primary Containment:** Flight approved Lexan sample container. The glove box will provide a second level of containment. (Preliminary discussion with the JSC toxicologist indicated two levels of containment are needed for the ZBLAN samples.)

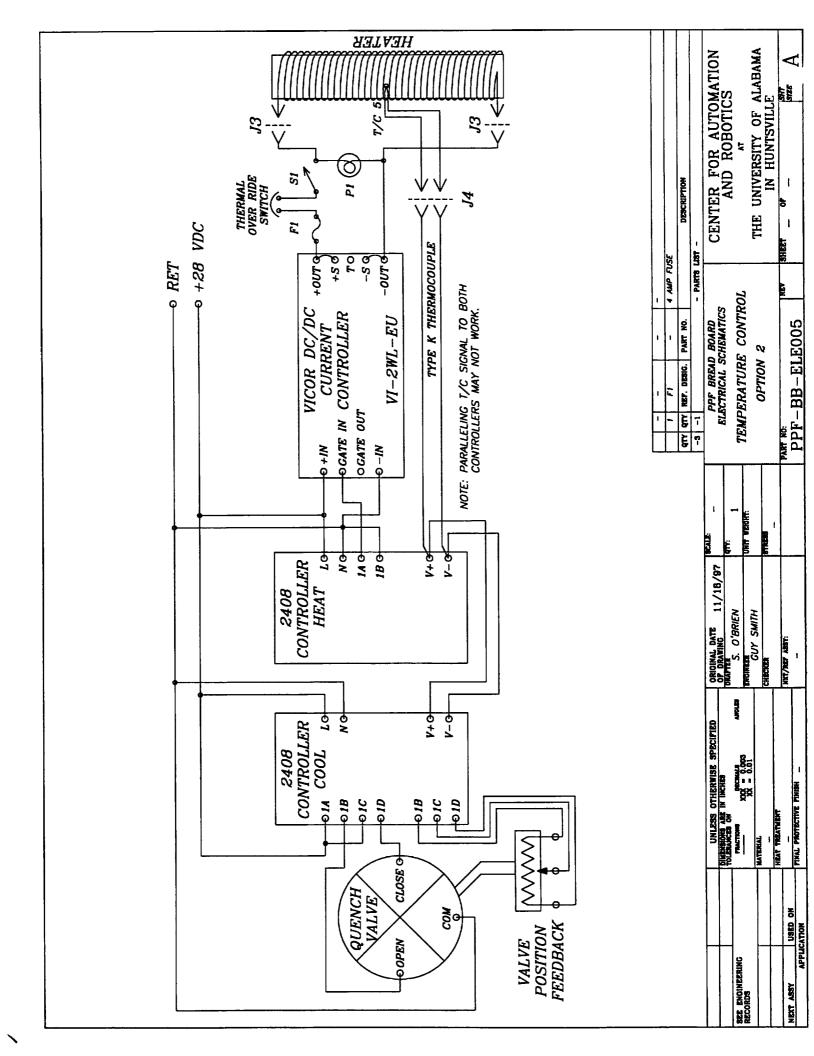
# ATTACHMENT 1

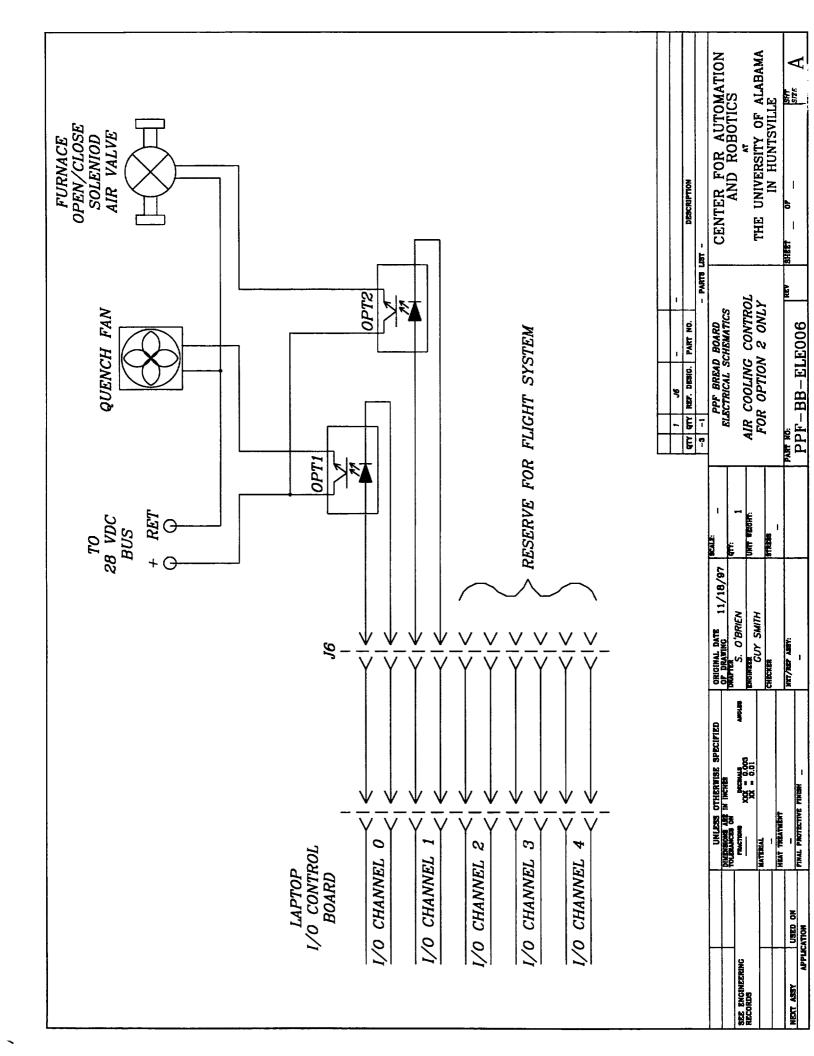
**Preform Processor Furnace Prototype Electrical Schematics** 

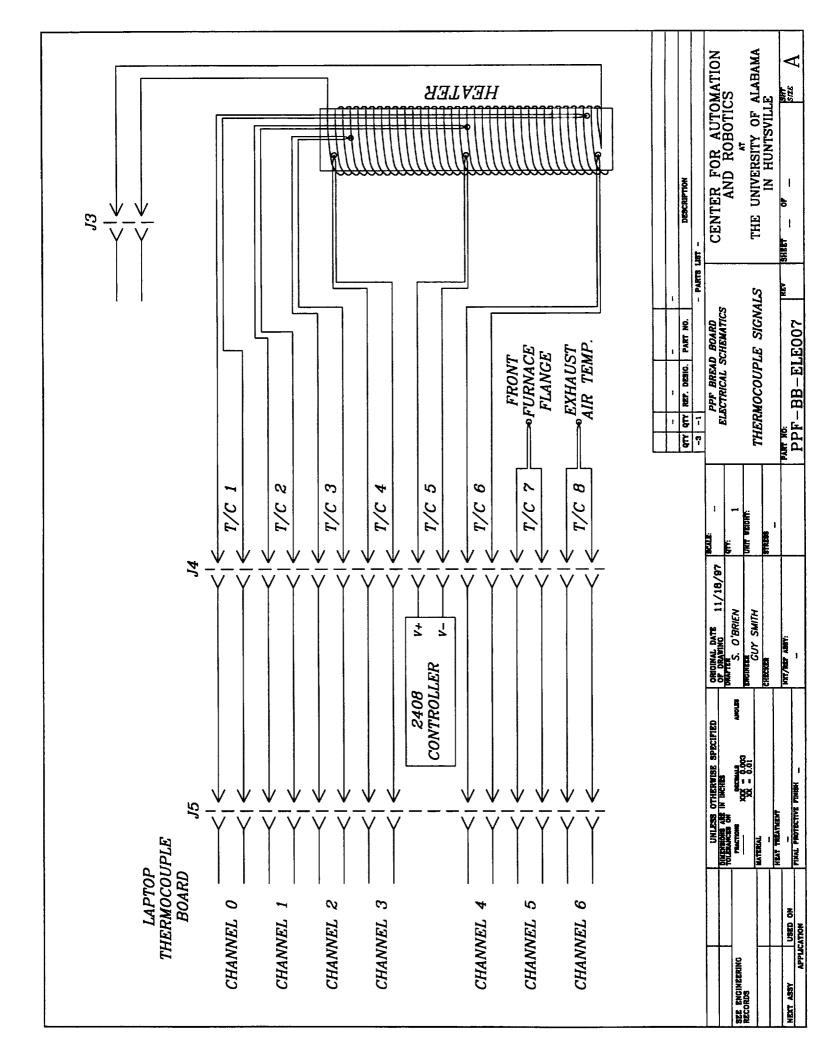












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## **Final Report**

#### Submitted to

# NATIONAL AERONAUTICS AND SPACE ADMINISTRATION GEORGE C. MARSHALL SPACE FLIGHT CENTER, ALABAMA 35812

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for Contract NAS8 - 97095, H-28163D Task 2

entitled

### Fiber Pulling Apparatus

by

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#### 1.0 INTRODUCTION

The fiber optics industry has grown into a multi-billion marketplace that will continue to grow into the 21st century.[1-5] Optical fiber communications is currently dominated by silica glass technology. Successful efforts to improve upon the low loss transmission characteristics of silica fibers have propelled the technology into the forefront of the communications industry. However, reaching the theoretical transmission capability of silica fiber through improved processing has still left a few application areas in which other fiber systems can provide an influential role due to specific characteristics of high theoretical transmission in the 2 - 3 micron wavelength region.

One of the other major materials used for optical fibers is the systems based upon heavy metal fluoride glass (HMFG). Commercial interest is driven primarily by the potential for low loss repeaterless infrared fibers. An example of the major communications marketplace which would benefit from the long distance repeaterless capability of infrared fibers is the submarine cables which link the continents.[5]

When considering commercial interests, optical fiber systems provide a healthy industrial position which continues to expand.[1-6] Major investments in the systems used for optical fiber communications have continued to increase each year and are predicted to continue well into the next century. Estimates of 8.5% compounded annually are predicted through 1999 for the North American market and 11% worldwide. The growth for the optical fiber cable itself is expected to continue between 44 and 50 per cent of the optical fiber communications budget through 1999. The total budget in 1999 world-wide is expected to be in the neighborhood of \$9 billion.[3] Another survey predicts that long haul telecommunications represents 15% of a world-wide fiber optics market in 1998.[4] The actual amount allotted to cable was not specified. However, another market research had predicted that the cable costs alone represents more than 50% of the total budget each year through 1998.[2]

A newly emerging activity is the commercial development of doped optical fibers which can be pumped by laser diodes to provide amplification of the communication signals.[6] This technology is newly emerging and will be developed for commercial interests in the United States by Galileo Electro-optical Incorporated in Sturbridge, MA on a license from British Telecom. Long repeaterless communication links provide the biggest stimulus for this technology. As an example of the of the revenues involved in the optical fiber communications

industry, the current trade journal lists that for the fiscal years, 1991 - 1994, 185 separate undersea links were established. In addition, another 105 links are planned through 1998. The distribution of revenues involved in the undersea installations is roughly \$8.5 billion through 1993 and another \$13 billion planned through 1998. A large portion of the future activity (34%) is planned for Southeast Asia and the Pacific Region. Other examples of the commercial utility of optical fiber networks is given in a recent scientific symposium in which the outlook for HMFG infrared fibers was determined to be very bright.[7]

Another area of interest lies in the use of fiber optics for laser surgery delivery systems, in which an optimal match between laser wavelength and fiber transmission characteristics occurs. For precise removal of tissue during surgery, research has shown that a wavelength in the 2.5 - 3.0 microns performs best. Experience with the combination of a pulsed Er: Yag laser (2.9 microns) delivered through a ZBLAN fiber has shown that this combination allows the removal of both fibrous and heavy calcified arterial plaque with little or no signs of thermal damage.[8] The 2.9 micron radiation corresponds quite closely with maximum tissue absorption (about 20 times greater than the 10.6 radiation from carbon dioxide lasers) and consequently allows very small penetration depth and precise tissue removal with no charring. This activity has proven to be of commercial value to small entrepreneurial companies such as Infrared Fiber Systems Inc. in Silver Spring, MD.

Process improvements which can enable heavy metal fluoride fibers to meet the their theoretical capabilities will provide the communication and medical industries with very desirable technology and products. Current manufacturers are very small and growth would be expected, as well as technology spin-offs to other manufacturers. It is the goal of space based experiments to provide much higher quality fiber with near theoretical transmission capability for development of commercial markets in the United States.

#### 2.0 BACKGROUND

The initial research into the use of multicomponent fluorides based on ZrF<sub>4</sub> chemistry led to the 1974 discovery of an amorphous product by Professor M. Poulain and his co-workers a the University of Rennes in France.[9] The remarkable feature of this work was that all the starting materials were crystalline and with the proper stoichiometry, amorphous glasses could be

produced. From this work, and others beginning to take an interest in the United States, the chemistry of the heavy metal fluoride glasses began to take shape.

An example of the HMFG chemistry is shown in Figure 1 below. The phase diagram for the ZrF4<sub>4</sub>-BaF<sub>2</sub>-LaF<sub>3</sub> (or ZBL glass) shows a region in which stable glass and a region of unstable glass devitrifies into crystalline material. An understanding of this chemistry led to the conclusion that ZF<sub>4</sub> was the glass former, BaF<sub>2</sub> was the modifier and a small amount of LaF<sub>3</sub> helped to decrease the devitrification rate was called a stabilizer. Other HMFG compositions were tried in the 1980's but none were as prolific in the research laboratories in both Europe and the United States as the ZBL systems. Further research evolved into the material of interest today, ZBLAN, in which AlF<sub>3</sub> and NaF are added to improve the glass forming capabilities of the final product. Current industrial practices uses hafnium for the cladding and consequently HfF<sub>4</sub> has also become an important ingredient in the overall chemistry.

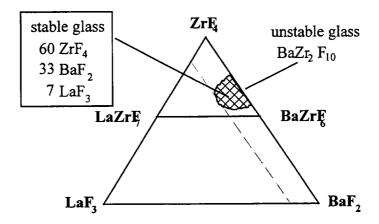


Figure 1. Phase diagram of typical HMFG system.

The primary focus on HMFG as an optical fiber material, in spite of its early shortcomings in reliable glass forming due to devitrification, was the theoretical prediction of low-loss of 0.001 dB/km at 3 microns as compared to silica (0.2 km/dB at 1.55 microns). The original theoretical work which indicated that the HMFG materials would have very good transmission characteristics was published by S. Shibata, et. al. and extended in more depth by the work of Lines, however equations defined in his work show the following result in Figure 2.[11]

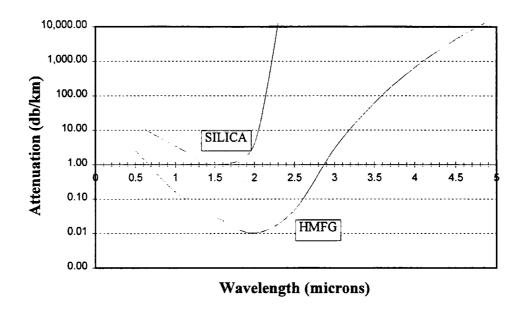


Figure 2. Comparison of theoretical attenuation for HMFG with average grade silica fibers.

The lower loss potential for long distance repeaterless fiber optic cable systems (undersea) and special purposes (medical laser surgery) promised a potential market of a high magnitude. For this reason many industrial and government laboratories world-wide began research in the HMFG including, Nippon Telegraph Telephone, British Telecom Labs, Naval Research Labs, Rome Air Development Center, ATT Bell Labs, Furukawa Industries, Corning Glass, Centre National d'Etudes des Telecommunications, and many more.

A number of formulations have been used to fabricate optical fiber devices in the 1980's and 90's. The use of zirconium fluoride as the primary constituent with fluorides of other metals such as barium, aluminum, sodium, lanthanum, etc. has led to general fiber compositions such as illustrated in Figure 3 below. The acronym used to describe these materials is based upon the metallic elements used in the fiber.

Acronym	ZrF4	$BaF_2$	LaF <sub>3</sub>	$AlF_3$	NaF	$T_{g}$	$T_x$	$T_c$
ZBLAN20	53	19	5	3	20	263	384	405
ZBLAN8	55	30	3	4	8	295	381	401
ZBLA	60	30	3	5		312		384
ZB	65	35				295	352	365

Figure 3. Typical formulations used for HMFG optical fibers.[12]

The major difficulties in ZBLAN processing have been determined to be due to the development of microcrystallites during both preform casting and during draw.[13-16] A major improvement in both the transmission and strength of the fiber can be obtained through a fiber fabrication process in which the microcrystallite population is substantially reduced.

Several research activities in microgravity science have observed the decreasing tendency of crystallization while processing in the microgravity environment.[18,19,21] The rationale for this behavior has not been determined at this time and continues to be a concept requiring further research. In the meantime, the industrial uses for an improved ZBLAN fiber provides a need to obtain a microgravity processed material in order to provide a better product of both industry and medicine.

### 3.0 CONTINUING WORK WITH THE GLASS ANNEALING FURNACE

In February of 1997 efforts turned to reflying the KC-135 Glass Annealing Furnace (GAF) as part of the continuing research efforts into the crystallization of ZBLAN fibers. During the previous flights of the GAF the hardware package was mounted in a manner in which the long axis of the fiber sample was perpendicular to the gravity vector. In many cases (during previous flights) this seemed to cause the fiber to "stick" to the inside wall of the sample ampoule and thus interfere with the results. Figure 4 clearly shows the effect on the ZBLAN

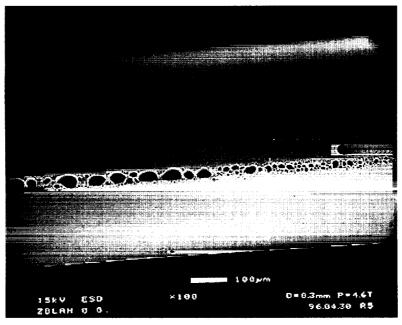


Figure 4: Surface reaction effects of ZBLAN with silica.

fiber reacting with the silica in the ampoule wall. This reaction causes the pitting as seen in the SEM photograph. To help alleviate this problem for the upcoming flight the package was remounted 90 degrees from its original position. This would allow the fiber a greater chance of avoiding full contact with the ampoule wall during the high-g period of the maneuver. Because this was considered a major change in the hardware the necessary safety documentation had to be prepared and submitted to JSC. No problems were encountered during this paperwork phase.

## 3.1 GAF Flight and Ground Results

During the week of April 21, 1997, the GAF was flown on NASA's KC-135 to process samples provided by Dr. Rifik Kortan of Lucent Technologies. Dr. Kortan was responsible for all sample preparations, which included drawing the ZBLAN fiber and loading the samples into the quartz ampoules.

During the first flight on April 22 no samples were processed. Only calibration tests were performed to verify the thermal characteristics and stability of the furnace in the various gravity levels. The results of these tests in the following table provided the verification that the furnace thermal characteristics were stable. Test Series 1 indicates the thermal stability of the preheat zone during the change from high-g too low-g.

TEST SERIES 1: PREHEAT ZONE STABILITY				
DURING CHANGING G-LEVELS				
PARABOLA	TEMPERATURE IN	TEMPERATURE AT		
NUMBER	нісн-с	END OF LOW-G		
1	255.1 C	254.3 C		
2	254.2 C	254.5 C		
3	254.2 C	253.9 C		
6	254.7 C	253.8 C		
7	254.3 C	254.2 C		
8	253.6 C	254.7 C		

Test Series 2 indicates the change in temperature the sample experienced during the transition from a stable temperature in high-g to the temperature reached at the end of low-g.

TEST SERIES 2: TRANSITION FROM PREHEAT						
zo	ZONE TO ANNEALING ZONE					
PARABOLA	ARABOLA TEMPERATURE IN TEMPERATURE AT					
NUMBER	HIGH-G	END OF LOW-G				
9	254.3 C	356.6 C				
11	252.4 C	355.3 C				
14	252.8 C	362.4 C				
16	251.3 C	358.3 C				
24	253.4 C	361.6 C				

Test Series 3 indicates the thermal stability of the annealing zone furnace during the change from high-g to the end of low-g. As indicated by the table the thermal stability is remarkably constant.

TEST SERIES 3: ANNEALING ZONE STABILITY  DURING CHANGING G-LEVELS						
PARABOLA						
NUMBER 17	HIGH-G 363.3 C	END OF LOW-G 363.4 C				
18	363.5 C	363.4 C				
25	362.1 C	362.3 C				

During all the thermal tests the preheat zone setpoint was at 240°C and the annealing zone setpoint was at 350°C. A fine gauge type K thermocouple placed inside an empty quartz ampoule was used to collect the thermal data and therefore simulated the thermal mass of a loaded quartz ampoule.

On the following day, during the second KC-135 flight, a total of nine ZBLAN loaded ampoules were processed in the GAF. Prior to processing the first ampoule the first five parabolas were used to calculate an average low-g time period. From the start of low-g to the end was found to be 24.3 seconds. This number was used to determine the maximum soak time of 21 seconds for the sample while in the annealing zone furnace. The initial preheat and annealing zone temperature setpoints were set to 240°C and 350°C, however the annealing zone temperature was reduced to 345°C after processing the first sample. It was determined that the sample melted too much at 350°C. The 345°C setpoint was used for all of the subsequent processing runs. The following table provided a summary of the flight days events.

3	SUMINARY OF APR	IL 23, 1997 KC-135 GAF PROCESSING RUNS		
PARABOLA NUMBER	SAMPLE I.D.	NOTES		
8	ZBLAN-302-1, #1	Sample melted too much, setpoint to 345, approx. 3 min. preheat		
12	ZBLAN-302-1, #2	Sample looked good, approx. 9 min. preheat period		
16	ZBLAN-302-2, #1	Sample looked good, fiber stuck to ID wall, approx. 2.5 min. preheat		
20	ZBLAN-302-2, #2	Sample looked good, approx. 2.7 min. preheat period		
22	ZBLAN-302-3, #1	Sample looked good, approx. 3.7 min. preheat period		
26	ZBLAN-302-3, #2	Large melt droplet on end of fiber, approx. 2.5 min. preheat period		
30	ZBLAN-302-3, #3	Sample looked good, approx. 2.6 min. preheat period		
32	ZBLAN-302-4, #1	Sample looked good, fiber stuck to ID wall, approx. 2.1 min preheat		
36	ZBLAN-302-4, #2	Small fiber diameter, approx. 3.0 min. preheat period		

On the following day, April 24, another seven samples were processed in the GAF system. Again the first five parabolas were used to calculate the average length of low-g which was determined to be 24.3 seconds. The annealing zone soak times were therefore limited to 21 seconds as before. Sample ID ZBLAN-302-4, #2 was processed twice, once on April 23 and again on the 24<sup>th</sup> by accident. The last sample, ZBLAN-302-6, #3 was processed three times during three successive low-g periods. The sample was retracted back into the preheat zone just prior to entering high-g.

PARABOLA NUMBER	SAMPLE I.D.	NOTES
9	ZBLAN-302-4, #2	Processed twice (once yesterday), approx. 2.4 min preheat period
13	ZBLAN-302-5, #1	Sample looked good, approx. 2.6 min. preheat period
18	ZBLAN-302-5, #2	Sample looked good however fiber stuck to ID wall, approx. 4.5 min PH
21	ZBLAN-302-5, #3	Sample looked good, approx. 4.0 min. preheat period
25	ZBLAN-302-6, #1	Sample looked good, approx. 3.0 min. preheat period
29	ZBLAN-302-6, #2	Sample looked good, approx. 3.0 min. preheat period
35, 36, 37	ZBLAN-302-6, #3	Processed three times, fiber reformed into two small BB's, approx. 10 mi preheat total

Ground runs were preformed in July 1997, with a total of six samples processed. All conditions (with the exception of gravity) were the same as those used during the flight runs. The preheat zone soak times were held constant to 3.0 minutes for each sample and the soak times in the annealing zone were held to 21 seconds. Only one sample was not processed either as a flight or ground sample and was used as a control or "as received" sample.

The following microphotographs provide a representative comparison between the flight and ground processed samples. The ampoules were broken open to remove the ZBLAN fibers for better photography. After photographing they were then replaced into the ampoules and taped closed. The fibers were photographed with polarized light to highlight surface crystals at a magnification of 56X.

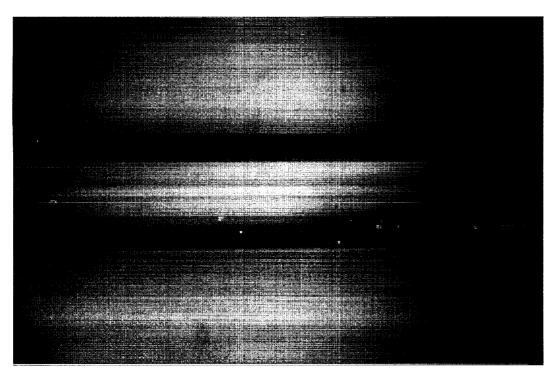


Figure 5: "As-received" control ZBLAN sample – not processed. The small white particles on the surface of the fiber are ambient dust and ZBLAN particles created when breaking the fiber to the proper length.

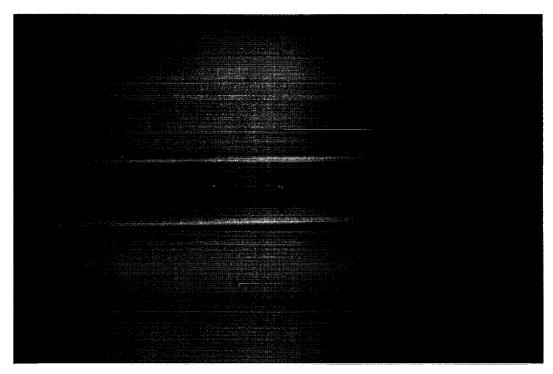


Figure 6. Ground processed sample ZBLAN-302-1, #3. Notice the "frosted" appearance of the fiber surface.

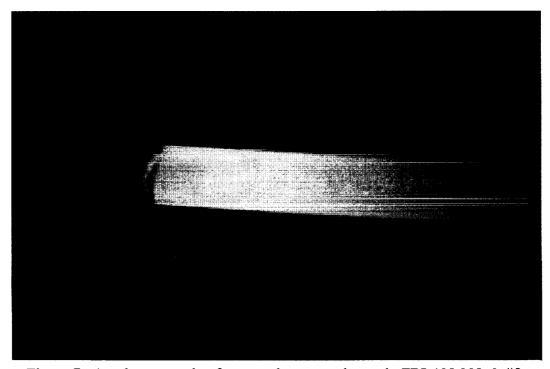


Figure 7. Another example of a ground processed sample ZBLAN-302-6, #5. In this case the surface crystallization is even more prevalent

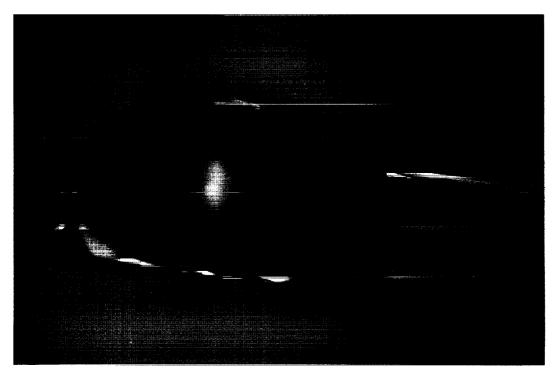


Figure 8. Flight processed sample ZBLAN-302-1, #1. Notice the lack of any evident surface crystallization. This sample is representative of all the flight processed samples.

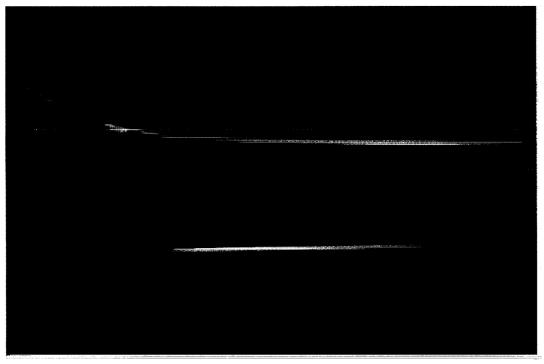


Figure 9. Another view of flight processed sample ZBLAN-302-1, #1

#### 3.2 Conclusion of GAF Results

During the April 1997 KC-135 flight of the GAF system some important information was obtained in regards to the thermal consistency of the two furnaces in low-g vs. high-g. In reviewing the thermal data it appears that there is little if any change in the thermal profile of either the preheat or annealing zone furnace when comparing the high-g data to the low-g data. This is important in that it indicates the ZBLAN fiber sample experiences the same thermal conditions whether it is processed in low-g or high-g. Since the thermal measurements took place over the gravity range of high and low is can be assumed that the thermal consistency is the same between low-g and normal-g. It can therefore be concluded that the crystallization phenomenon, or lack thereof, is not due to differences in the thermal processing parameters (i.e. convection) in the GAF but due to the differences in gravity.

The presented microphotographs provide good correlation to previous GAF experiments results [22, 23]. It can be clearly seen that ZBLAN samples processed in a normal 1-g environment develop microcrystallites on the surface of the fiber where as flight processed samples in low-g did not. In the ground processed samples (six in all) two of the samples (ZBLAN-302-1, #3 and ZBLAN-302-6, #5) showed almost complete surface crystallization. The remaining four ground samples processed had only about 5 to 10 percent crystallization. The reason why is as yet undetermined at the time of this printing. It is suspected that differences in how the samples were prepared and sealed inside the ampoules may be the reason. Work is currently underway at UAB to provide SEM and TEM microphotographs of the samples presented in this report.

# 4.0 SUPPORT EFFORTS ON THE PPF PROTOTYPE

In the spring of 1997 the MSFC Space Sciences Labs began development of a Preform Processor Furnace prototype. This furnace is intended as an engineering test bed for developing the Shuttle MidDeck version. The purpose of the PPF is the processing ZBLAN preforms in the microgravity environment of earth orbit. In view of the results obtained from the GAF experiments it is a strong possibility that earth based manufactured ZBLAN preforms which contain microcrystallites can be reheated to 800°C in microgravity, dissolve out the defects, and then quench the preform back into a solid rod. The preforms would then be returned from space and distributed to the industrial partners who would then draw the ZBLAN fiber. It is expected that by starting out with a preform free of microcrystallites a nearly perfect optical fiber can be produced.

General design support was provided on a weekly basis in the development of PPF prototype furnace. This support included identification of temperature and power control subsystems and parts which could be readily obtained from commercial sources and subsequently used (with minor modifications) in the actual flight hardware system. Electrical schematics were developed and provided to the SSL team in charge of assembling the prototype system. Copies of these drawings are included in Attachment 2 at the end of this document. In addition, other areas of general support were also provided including thermal design assessments, Shuttle and astronaut interface requirements, safety, sample cartridge designs, and data acquisition/control issues.

#### 5.0 EARLY DEVELOPMENT OF MAGE

In addition to the GAF activities an additional task was taken on to develop a Space Shuttle experiment which could prove that ZBLAN could be held in a magnetic field and then processed in the same manner as GAF samples. In this way ZBLAN could be processed in a containerless environment and over a longer period of time. This would provide additional data points into the growth kinetics of the microcrystallites. Early in October of 1997 work began on developing such an experiment for flight on STS-95 in October 1998. Prior to that, a simpler demonstration package was developed for testing out the theory on the KC-135. This was a collaborative effort between UAH and Boeing. The concept for the levitating the ZBLAN (or any other diamagnetic material) was developed by Boeing [24].

The purpose of MAGE (Magnetic Glass Experiment) was to demonstrate the levitation of ZBLAN glass in a magnetic field. ZBLAN glass, like many other materials, is diamagnetic. In the absence of gravity this property allows materials to be suspended in a magnetic "bottle" between two opposing permanent magnets.

### 5.1 MAGE Experiments on the KC-135

Experiment hardware was designed to demonstrate magnetic levitation on board the NASA KC-135 reduced gravity aircraft. Two separate weeks of flights were made. Each flight week was four days, with about 40 parabolas each day. Each parabola provided about 25 seconds of reduced gravity. To further reduce the gravity level, the experiment package was allowed to free float at the end of an umbilical consisting of power and data cables. The free float resulted in brief periods of low gravity on the order of 1 to 2 milli-g. These lasted from 2 to 5 seconds. A camera on the free float package allowed a video tape record of each flight.

The first flight week was July 12 to 17, 1997. The magnet/sample holder assembly that was flown is shown in figure 10.

The sample containers for this flight were glass cylinders attached to the end of the sample holders. Two sample materials were flown: ZBLAN glass and Bismuth. Bismuth was chosen because it is the most diamagnetic material and therefore is most affected by the magnetic field. The samples were in an air environment within the sample container. During the first flight day it was observed that if the samples were subjected to an initial disturbance they never became stable within the container. Any small motion resulted in the sample bouncing off

the walls of the container similar to a "ping pong" effect. Another problem was the adhesive used to seal the containers did not fully cured and as result remained slightly tacky. This allowed the samples to stick in the corners. These problems resulted in the early withdrawal of the experiment from the aircraft after two days.

The second KC-135 flight took place the week of December 8, 1997. The hardware was redesigned to accommodate larger, more powerful magnets. The magnet holder was made from acrylic blocks. Two one inch diameter by ½ inch thick magnets and two-½ inch diameter by ¼ inch thick magnets were mounted on either side of a gap that formed the magnetic "bottle". A drawing of the magnet holding assembly is shown in figure 11. Clear plastic sample containers were used for the samples in air. These had a foam pad at one end for damping. Plastic vials were used for containers in which a liquid solution was used to provide viscous damping. The best results were obtained with this approach. The ZBLAN was suspended between the magnets on several occasions for short durations. The undisturbed levitation time was on the order of two to three seconds.

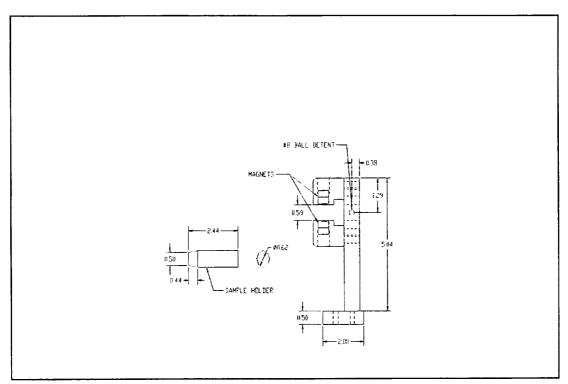


Figure 10: Side view of first prototype magnetic levitator (mirror not shown).

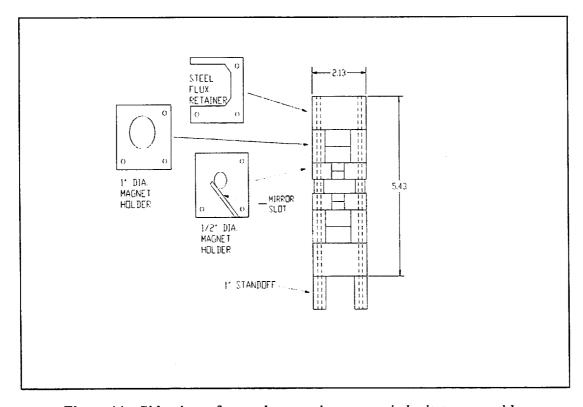


Figure 11: Side view of second generation magnetic levitator assembly.

## 5.1.1 Summary of Accelerometer Data from MAGE

The following accelerometer data is a representative sample of typical free float periods for the MAGE hardware during KC-135 low-g maneuvers. Each data point is an average of

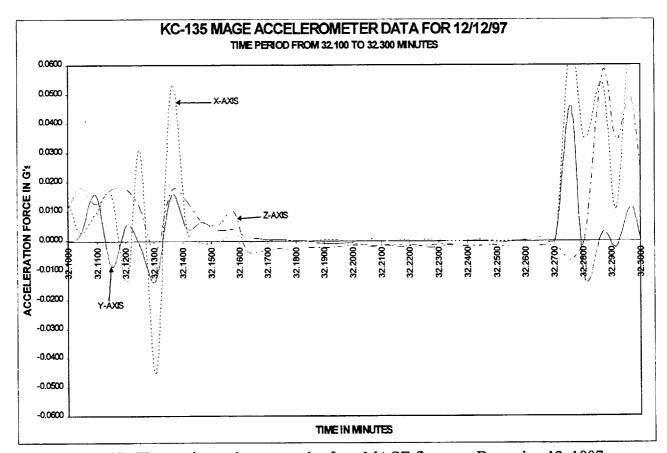


Figure 12. Three axis accelerometer plot from MAGE flown on December 12, 1997

1000 readings which filters out almost any EMI noise present on the accelerometer signals. This data was collected during the December 12, 1997 KC-135 flight day. The following captured video images (Figures 13 and 14) are of a ZBLAN sample being held within the magnetic bottle during the low-g period shown in the above graph. The ZBLAN sample was rectangular shaped 1.5x1.5x3 mm in size. The sample cell was filled with an ethanol/MgCl mixture that had the same magnetic properties as air.

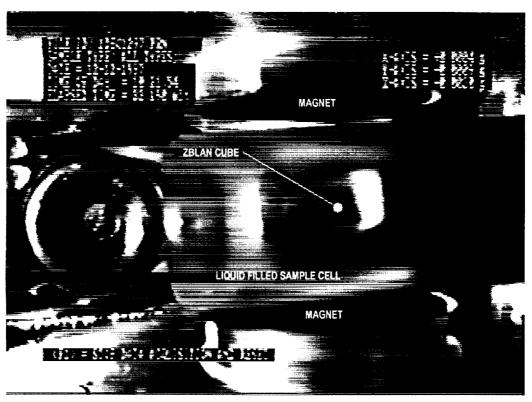


Figure 13. Video image of a ZBLAN cube suspended in the magnetic bottle region of MAGE.

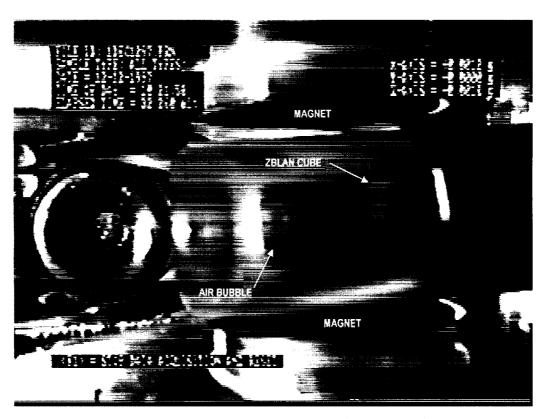


Figure 14. Image of the same cube floating out of the magnetic bottle region due to the negative 0.002 milli-g acceleration in the Z-axis.

#### 5.2 Shuttle/SpaceHab Glovebox Experiment

The previous experiments were precursors to designing an experiment to fly on the Shuttle on STS 95 scheduled for October 1998. The experiment is being designed to be carried out in the Microgravity Glovebox (MGBX) to be mounted in the SpaceHab module. A visit was made to the MGBX facility at Marshall Space Flight Center (MSFC) to become familiar with the features and accommodations offered. There are two items supplied that can be utilized: a video camera and a light source. These items and others were measured to allow inclusion in the design. The measuring was necessary since there are no dimensioned drawings available for the Glovebox or its accessories. An Interface Control Document (ICD) has not been developed, so users must make their own measurements and obtain other information on connectors, power sources, etc.

A conceptual design of the hardware has been completed. A set of the drawings is attached. A request has been made be Boeing to include additional equipment and temperature sensors to perform measurements of magnetically driven convection of heated air. These requirements have not yet been incorporated into the design.

#### 5.2.1 Materials List

The following are the materials that have been identified for use in the Shuttle experiment hardware. The list should not be considered to be complete or final.

- 1. Aluminum
- 2. Flight approved polycarbonate
- 3. Stainless steel
- 4. Sheet steel (magnetic shielding)
- 5. Silica glass (sample container)

#### 5.3 Attachment 1

This is the Sample Material Submittal that was sent to Amy Haas, the experiment Payload Coordinator at SpaceHab (STS-95) on 11/17/97. This information was provided as part of the initial safety documentation.

**Experiment:** Magnetic Levitation of ZBLAN Glass (MAGE)

PI: Dr. Dennis Tucker, NASA, 205-544-2685

Co-PI: Dr. Brian Tillotson, Boeing, 253-773-4547

Hardware development: Leonard Adcock, UAH, 205-890-6086 x 225

**Experiment Summary:** The experiment will be carried out in the Glovebox. ZBLAN glass is diamagnetic. In the absence of gravity it can be levitated magnetically using two sets of permanent magnets. The magnets are mounted opposed with a gap of approximately one half inch between them. The magnets are held in a holder designed for the purpose. The samples, in their containers, are inserted into the gap and observed. The observation will be made using a video camera, recorded, and downlinked if possible.

#### **Test Materials:**

Material	Quantity	Weight	Volume
ZBLAN* glass	6	<10g ea.	~25 mm³ ea.
Silica glass	1	<10g ea.	~25 mm³ ea.
Lysozyme protein crystal in aqueous solution of NaCl	1	<10g ea.	~25 mm³ +2cc.
Bismuth	1	<10g ea.	~25 mm³ ea.
Water	1		~2cc
Wood	1	<10g ea.	~25 mm³ ea.
Plastic	1	<10g ea.	~25 mm³ ea.

<sup>\*</sup> ZBLAN is a heavy metal fluoride glass containing Zirconium fluoride, Barium fluoride, Lanthanum fluoride, Aluminum fluoride, and Sodium fluoride.

Weight of Materials: see table above.

**Test Conditions:** Five to ten minutes of video recording of each sample.

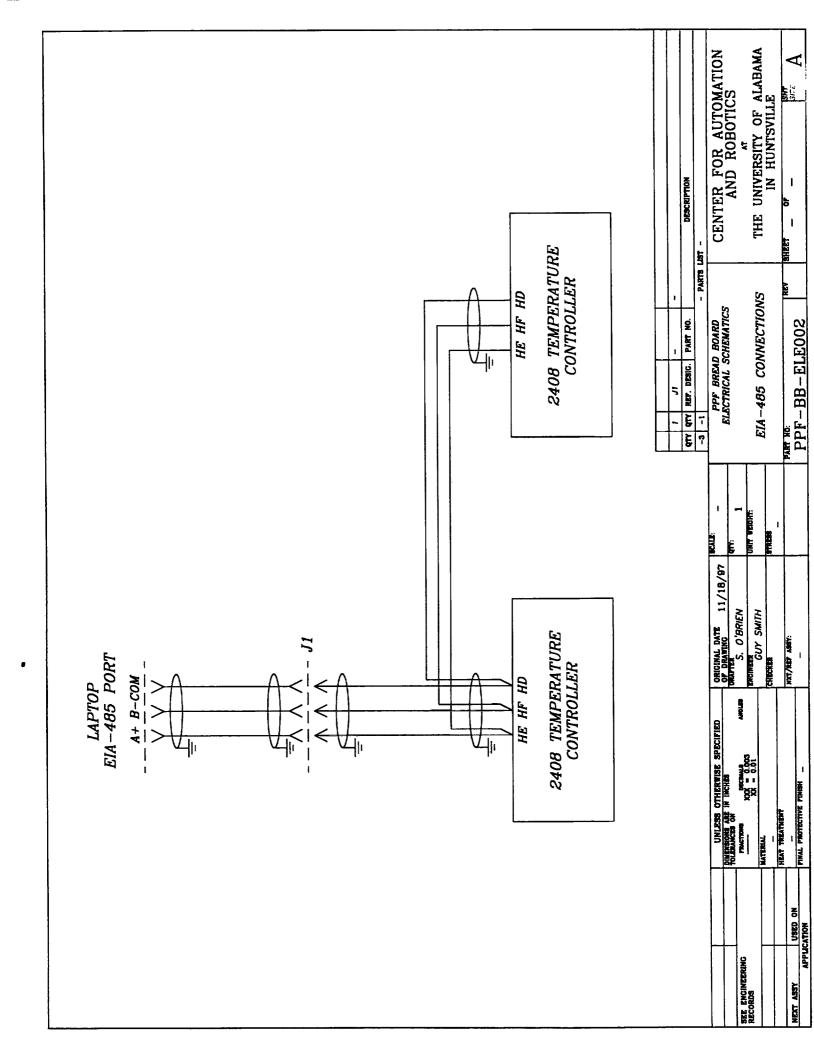
Number of Samples: 11

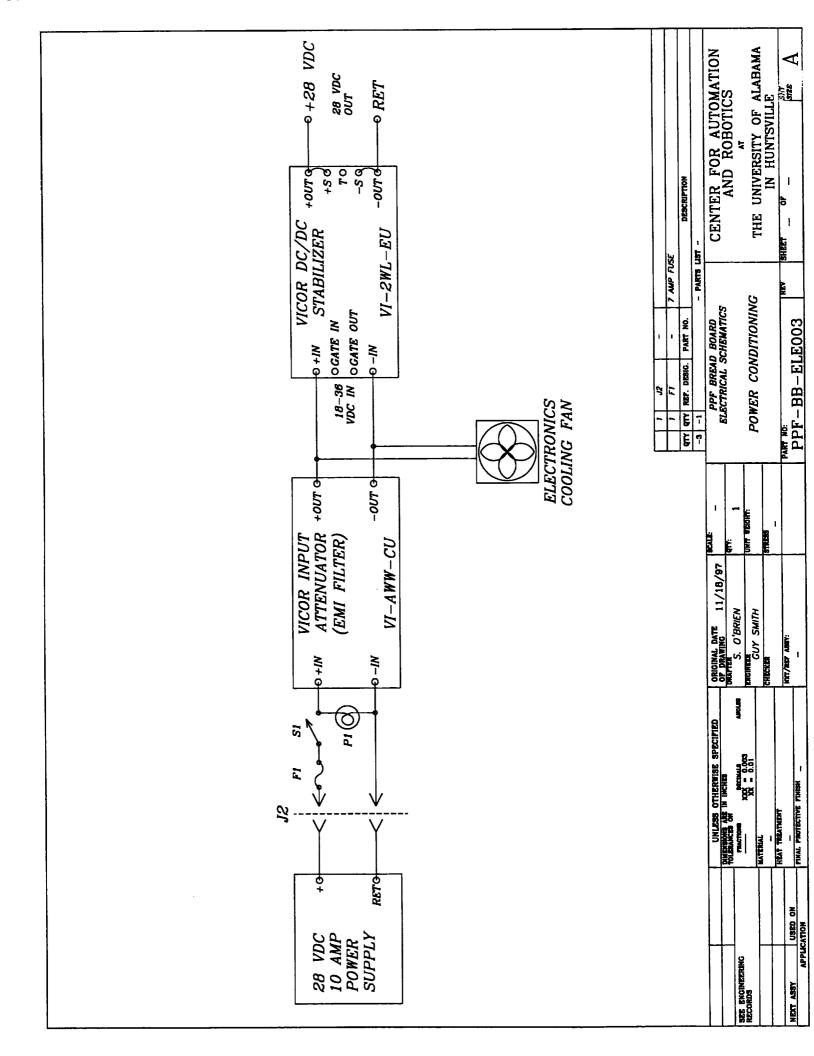
#### Types and Estimated Level of Toxic Hazard:

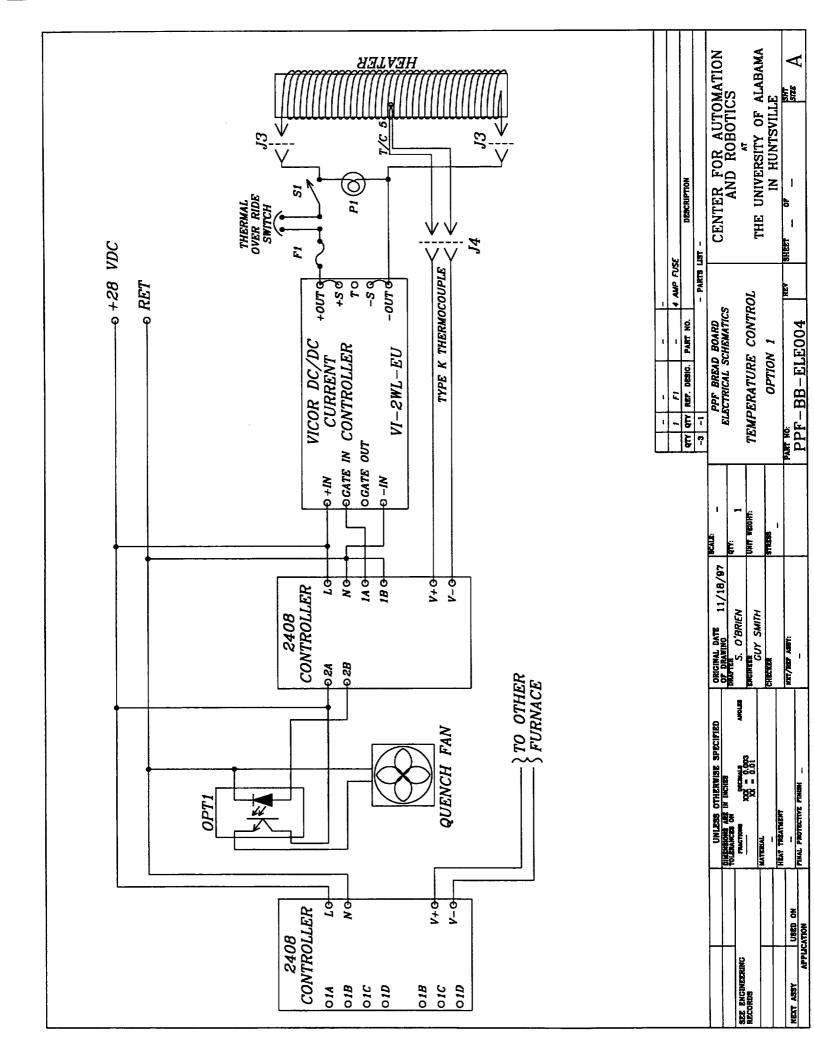
**Primary Containment:** Flight approved Lexan sample container. The glove box will provide a second level of containment. (Preliminary discussion with the JSC toxicologist indicated two levels of containment are needed for the ZBLAN samples.)

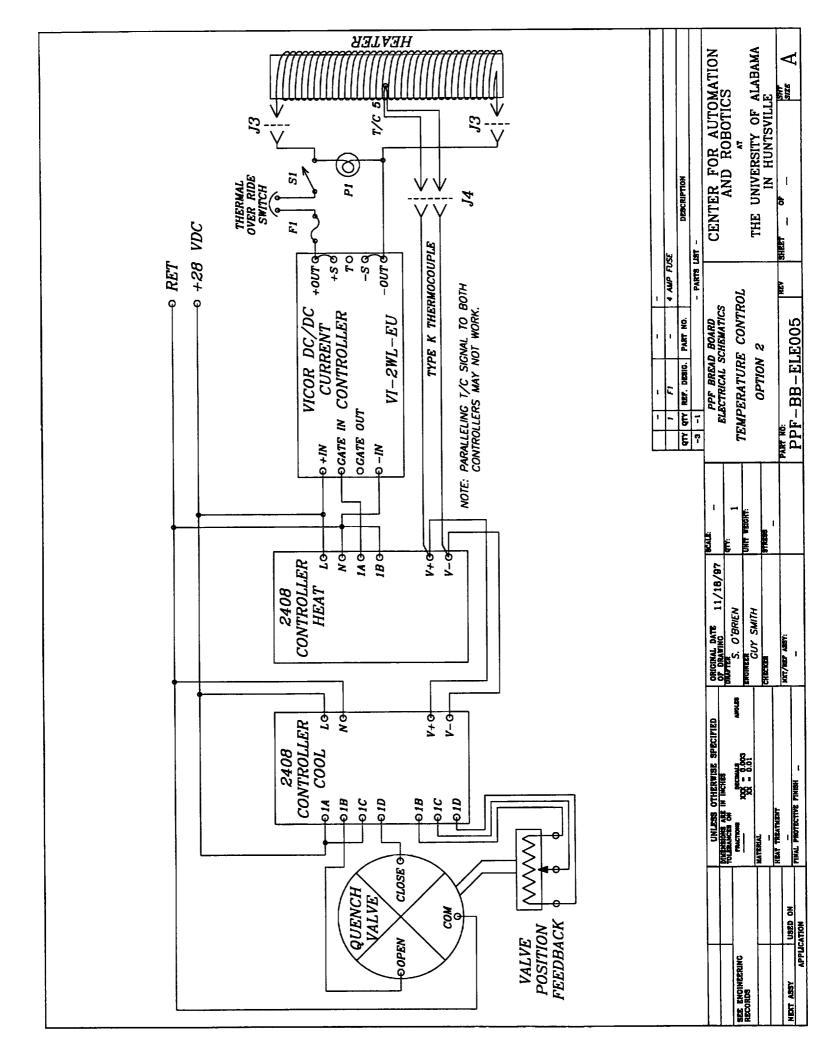
# **ATTACHMENT 1**

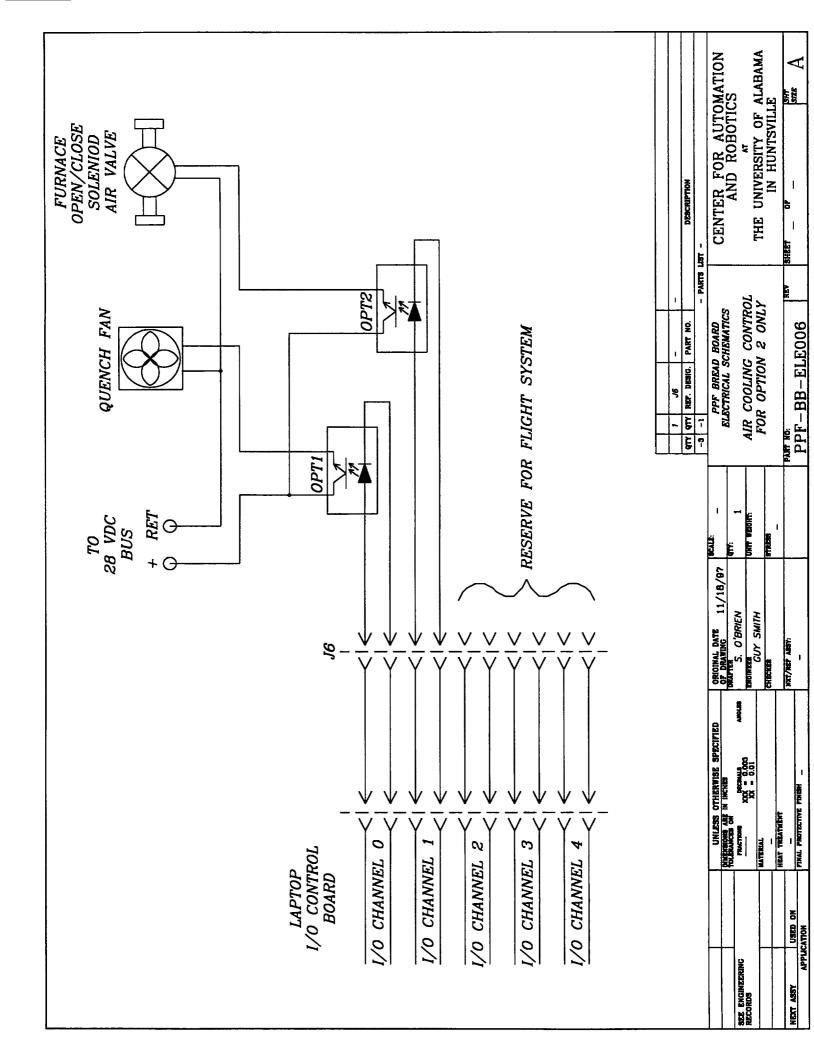
**Preform Processor Furnace Prototype Electrical Schematics** 

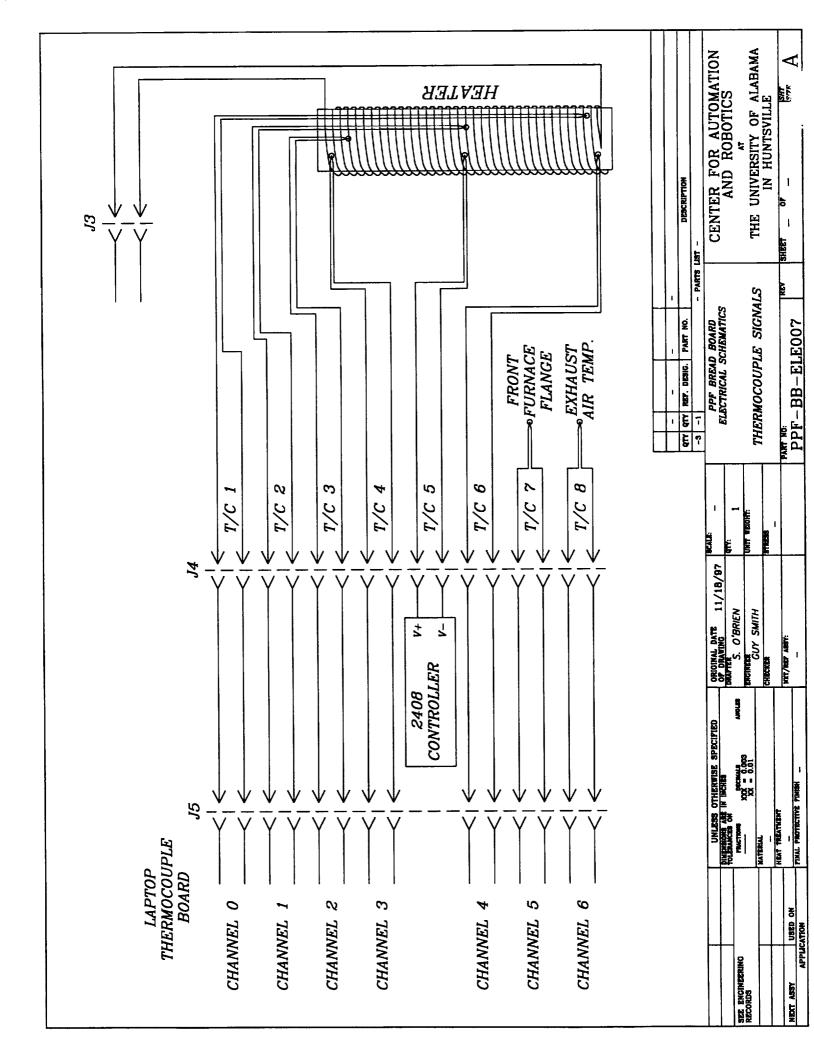












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